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(54) Title: HAIR CARE COMPOSITIONS (57) Abstract A hair care composition comprises an anti-free-radical agent and a polymeric conditioning agent prepared from monomer entities selected from one or more of the following: acrylic acid, acrylamide, dimethyldiallylammonium chloride and any mixtures thereof. Preferably the anti-free-radical agent is selected from one or more of ascorbic acid, tocopherol, a herbal extract (e.g. birch extract) and any mixtures thereof. Methods of preparing such compositions are described. Use of the hair care compositions in a method of treating hair (e.g. washing, conditioning and/or styling hair) is also described.		

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HAIR CARE COMPOSITIONS

The present invention relates to hair care compositions having conditioning properties, to methods of preparing such compositions and to methods of using such compositions to treat hair.

5 The term "hair care composition" as used herein includes so-called "hot oil" treatments, shampoos, conditioners, hair dyes, mousses, foams, gels, creams, waxes, masks, muds, styling sprays, lotions and rinses, all suitable for use on animals, preferably on humans, most preferably on the human head.

10 Hair care compositions having conditioning properties are well known in the art. For example, GB-A-1538768 (Alberto Culver) discloses a method of treating hair to improve its manageability, by contacting the hair with an aqueous composition comprising a water soluble quaternary ammonium compound (having one or two nitrogen atoms and at least one long chain alkyl group linked to one or both of the nitrogen atom[s]), and then shampooing the
15 hair.

Unfortunately, many conditioning agents tend to promote free-radical reactions in the composition on storage or in use. Such reactions generate free-radicals, especially on exposure to sunlight and/or heat, and presence of high concentrations of free radicals is generally seen as undesirable in a
20 toiletries composition and when used on the hair.

It has now surprisingly been found that certain types of conditioning agents have a greatly reduced tendency to promote free-radical reactions. Thus it has been found that by use of these conditioning agents in combination with an anti-free-radical agent, a composition can be provided which generates
25 surprisingly low levels of free-radicals in use.

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Free radicals, which are generated by factors such as UV radiation (present in sunlight), heat and/or by chemical reaction, are implicated in the process of damage to hair and fading of hair colour. The hair care compositions of the present invention have been shown to generate lower levels of free radicals than known hair care compositions. Therefore the hair care compositions of the present invention may be used to provide improved protection against the fading of natural or permanently coloured hair by sunlight and against damage to hair caused by exposure to factors such as sunlight, environmental and/or atmospheric pollution, heat from styling the hair and/or chemical treatment of the hair (for example curling, perming, straightening, dyeing and/or bleaching). The hair care compositions of the present invention may comprise conventional hair care products and/or specific hair protection products which may be used for example as protective pre-treatments prior to heat or chemical treatment of the hair.

Therefore broadly according to the present invention there is provided a hair care composition comprising an anti-free-radical agent and a polymeric conditioning agent prepared from monomer entities selected from one or more of: acrylic acid, acrylamide dimethyldiallylammonium chloride and any mixtures thereof.

Suitably, the polymeric conditioning agent is a copolymer of acrylic acid and acrylamide monomer entities, and may comprise further comonomers.

Preferably the polymeric conditioning agent is a polymer of dimethyldiallylammonium chloride monomer entities, more preferably a copolymer further comprising acrylamide comonomer entities, most preferably the copolymer still further comprising acrylic acid comonomer entities.

In one embodiment, the polymeric conditioning agent is a terpolymer comprising acrylic acid, acrylamide and dimethyldiallylammonium chloride

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comonomers. Suitably these comonomers are present in weight ratios of about 1 to about 2 to about 1 respectively.

Particularly preferred polymeric conditioning agents are the polymers known under the following names assigned by the Cosmetic Toiletries and
5 Fragrance Association (hereinafter known as CTFA designations):
polyquaternium 39' which is a terpolymer of dimethyldiallylammonium chloride, acrylamide and acrylic acid comonomers (available commercially from Chemvion Speciality Chemicals under the trade names 'Merquat Plus 3330'
and Merquat Plus 3331'); 'polyquaternium 7' which is a copolymer of
10 dimethyldiallylammonium chloride and acrylamide comonomers (available commercially from Chemvion Speciality Chemicals under the trade name 'Merquat S'); and 'polyquaternium 6' which is a homopolymer of dimethyldiallylammonium chloride monomers (available commercially from Chemvion Speciality Chemicals under the trade name 'Merquat 100').

15 The polymeric conditioning agent may be present in the composition in an amount of from about 0.001% to about 10% by weight, suitably from about 0.01% to about 5% by weight, for example about 1% by weight.

The anti-free-radical agent may be, for example ascorbic acid, vitamin E (tocopherol), lipacide LHL, phytoamine or herbal extract such as mimosa
20 tenuiflora AA (available commercially from A & E Cannock), ginkgo biloba extract (available commercially from Indena), or birch extract. Preferred anti-free-radical agents are selected from one or more of: ascorbic acid, tocopherol and herbal extract. Particularly preferred anti-free- radical agents are selected from ginkgo biloba extract and birch extract. Ginkgo biloba extract
25 may be produced by extracting the leaves of the ginkgo biloba tree with a suitable solvent. It is believed that the anti-free-radical activity of ginkgo biloba extract arises from the presence of flavonglycocides and/or terpenelactones which may be free-radical inhibitors. Birch extract may be produced by

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extracting the dried leaves of *Betula alba* with a suitable solvent. It is believed that the anti-free radical activity of birch extract arises due to the presence of flavonoids such as hyperosid, quercitrosid and/or myricetol-3-digalactosid which may be free-radical inhibitors. A suitable birch extract comprises those
5 available from Cosmetochem (U.K.) Ltd under the trade names "Super Herbasol Extract Birch" and "HP Herbasol Betula" and those available from Blagden Chemicals under the tradenames "Phytelene of Birch" and "Aqueous Spray Dried Birch".

The anti-free-radical agent may consist of active ingredients which are
10 free-radical inhibitors or may also comprise suitable diluents and/or carriers (such as when the anti-free radical agent is some of the products mentioned herein). It will be appreciated that the anti-free-radical agent comprising the present invention is not limited to one or more of those anti-free-radical agents listed above and is not limited to those anti-free-radical agents comprising the
15 ingredients described above.

Preferably, the anti-free-radical agent may be present in an amount of from about 0.01% to about 10% by weight, more preferably from about 0.05% to about 5% by weight, most preferably from about 0.15% to about 3.0% by weight of the composition.

20 To further reduce the generation of free-radicals by the UV radiation in sunlight, compositions of the present invention may further comprise any acceptable suncreening agent (that is an agent which acts to absorb and/or reflect UV radiation present in sunlight) and which would be acceptable for use in a hair-care composition (for example suitable for use on the human head).
25 Such suncreening agents may comprise inorganic sunscreens (for example zinc oxide and/or titanium dioxide preferably of microfine (< 100 nm) particle size) and/or organic sunscreens (for example p-aminobenzoic acids, esters and derivatives, methoxycinnamate esters, benzophenones [such as

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benzophenone-4 {available commercially under the trade name Uvinul MS40}}; dibenzoylmethanes and/or salicylate esters). The sunscreens agents may be present in an amount of from about 0.1% to about 10% by weight of the composition.

- 5 Further components may be added to the hair-care composition as is well-known to those skilled in the art.

For example, preservatives may be added to the composition such as 2-bromo-2-nitropropane-1,3-diol (bronopol, which is available commercially under the trade name Myacide), benzyl alcohol, diazolidinyl urea, imidazolidinyl
10 urea, methyl paraben, phenoxy ethanol, propyl paraben, sodium methyl paraben and sodium propyl paraben, suitably in an amount of from about 0.01% to about 10% by weight of the composition.

Thickeners and viscosity modifying agents may be added to the composition, such as amine oxides, block polymers of ethylene oxide and
15 propylene oxide (for example, those available from BASF Wyandotte under the trade name "Pluronic"), ethoxylated fatty alcohols, cellulosic derivatives such as hydroxypropylmethyl cellulose, salt (NaCl), phthalic acid amide, polyvinyl alcohols and fatty alcohols, suitably in an amount of from about 0.5% to about 10% by weight of the composition.

- 20 Sequestering agents may be added to the composition, such as ethylenediamine tetraacetic acid and salts thereof, suitably in an amount of from about 0.005% to about 0.5% by weight of the composition.

The composition may also include resins such as: octylacrylamide / acrylates / butylaminomethacrylate copolymer (available under the trade name
25 Amphomer); ethyl ester of polyvinylmethyl (hereinafter known as PVM) / methylacrylate (hereinafter known as MA) copolymer (available under the trade

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name Ultrahold 8A); vinyl acetate (hereinafter known as VA) / crotonates / vinyl
neodecanate copolymer (available under the trade name Adhesive 28-2930
NAL); acrylates / acrylamide copolymer (available under the trade name
Gantrez ES225); vinyl acetate / crotonic acid / vinyl propionate copolymer
5 (available under the trade name Luviset CAP); polyvinylpropionate (hereinafter
known as PVP) / VA / vinylpropionate copolymer (available under the trade
name Laviskol VAP); octylacrylamide / acrylate copolymer (available under the
trade names Versatyl 90 or Lovocryl 47); vinyl caprolactam / PVP /
dimethylaminoethyl methacrylate copolymer (available under the trade name
10 (H₂O LD EP-1); PVM / MA copolymer (available under the trade name Gantrez);
and vinyl acetate / butyl maleate / isobornyl acrylate copolymer (available under
the trade name Advantage CP). These resins may be present suitably in an
amount of from about 0.1% to about 10% by weight of the composition.

The composition may also include slip aids such as phenyl trimethicone,
15 suitably in an amount of from about 0.1% to about 10% by weight of the
composition.

The composition may also include vitamins such as biotin, suitably in an
amount of from about 0.01% to about 1.0% by weight of the composition.

The composition may also include waxes such as cocoa butter suitably in
20 an amount of from about 1% to about 99% by weight of the composition.

The composition may also include gelling agents such as PVM, MA, or a
decadiene crosspolymer (available under the trade name Stabizez 06), suitably
in an amount of from about 0.1% to about 2% by weight of the composition.

The composition may also comprise suitable, cosmetically acceptable
25 diluents, carriers and/or propellants such as dimethyl ether.

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The composition may also include pearlising agents such as stearic monoethanolamide, suitably in an amount of from about 0.01% to about 10% by weight of the composition.

5 Perfumes may be added suitably in an amount of from about 0.01% to about 2% by weight of the composition, as may water soluble dyes such as tartrazine, suitably in an amount of from about a trace amount (such as 1×10^{-5} %) to about 0.1% by weight of the composition.

The composition may also include pH adjusting agents such as sodium hydroxide, aminomethyl propanol, triethanolamine, suitably in an amount of
10 from about 0.01% to about 10% by weight of the composition.

The composition may be buffered by means well known in the art, for example by use of buffer systems comprising succinic acid, citric acid, lactic acid, and acceptable salts thereof, phosphoric acid, mono- or disodium phosphate and sodium carbonate. Suitably, the composition may have a pH
15 between about 3 and about 10, preferably between about 4 and about 8.

The composition may also include an antidandruff agent such as salicylic acid or zinc pyrithione suitably in an amount of from about 0.1% to about 5% by weight of the composition.

20 Surfactants may be included, such as cosmetically acceptable salts of alkyl ether sulphates, alkyl and alkylamidoalkyl betaines, ethoxylated alcohols, polyethyleneglycol carboxylates, acceptable salts of alkyl sulphates (such as ammonium lauryl sulphate), sulphosuccinates (such as disodium laureth sulphosuccinate), amphotoacetates and amphodiacetates (such as disodium cocoamphodiacetate), alkylpolyglucosides and alcohol sulphonates.

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Broadly in accordance with a further aspect of the present invention there is provided a method of preparing a hair care composition comprising mixing in a suitable manner an anti-free radical agent with a polymeric conditioning agent prepared from monomer entities selected from one or more of: acrylic acid, acrylamide, dimethyldiallylammonium chloride and any mixtures thereof. 5 Optionally any other suitable ingredients may be added such as those described herein. Preferred methods of preparation are described in the examples.

Broadly in accordance with a still further aspect of the present invention 10 there is provided a method of treating hair (for example washing, conditioning and/or styling hair) by application of a composition as described herein.

The invention will be understood with reference to the non-limiting tests and formulation examples described hereinafter:

Free-Radical Test Results

15 A variety of hair-conditioning agents were tested for effects on free-radical generation by use of a standard hypoxanthine / xanthine oxidase enzyme-based assay.

The assay involves reduction of tetrazolium nitroblue (NBT) into formazan blue by superoxide anions produced by the action of xanthine oxidase on 20 hypoxanthine. The formation of formazan from NBT creates a deep blue colour which is detectable by use of a spectrophotometer at 560 nm.

In the presence of oxygen free radical scavengers, the reaction is inhibited; the percentage inhibition indicates the efficiency potential of the "scavengers".

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The rate of formation of formazan blue in relation to time is known to be substantially linear during the first five minutes of reaction, the reductive activity of superoxide radicals thus being directly proportional to the rate of change of absorbance over time. Hence any reduction in this rate of change in the presence of any test material is proportional to oxygen free radical scavenging activity.

Reagents were made up as follows:

1. Tris/HCl buffer, 0.05 M, pH 7.4
2. Solution of xanthine oxidase in Tris/HCl buffer (1 above) at an activity of 1.67 Units/ml.
3. Solution of hypoxanthine in Tris/HCl buffer (1 above) at 0.5×10^{-2} M.
4. Solution of tetrazolium nitroblue in Tris/HCl buffer (1 above) at 10^{-3} M.

Assays were carried out in disposable plastic cuvettes. In each case 0.5 ml of solution 3 (hydroxanthine solution), 0.1 ml of solution 4 (tetrazolium nitroblue solution) and sufficient of solution 1 (buffer solution) to produce a total of 3 ml, were placed in a cuvette. 0.1 ml of test solution (a 2% w/w solution of a conditioning agent) was added followed by 0.1 ml of solution 2 (xanthine oxidase solution) and the absorbance of the resulting mixture was immediately followed using a UV/visible spectrophotometer at 560 nm.

Negative controls (without presence of xanthine oxidase or conditioning agents) and positive controls (with xanthine oxidase but without conditioning agents) were also carried out in which the cuvettes were made up as described above with the addition of 2.4 ml (negative control) and 2.3 ml (positive control) of solution 1 respectively, so the total liquid in each cuvette was also 3 ml. The percentage inhibition of free radical activity for each conditioning agent was then calculated relative to these controls. Results are set out in Table 1.

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As used herein the terms 'FRA' denotes free radical activity; '% change' denotes percentage change; '% w/v' denotes percentage ingredient (in grammes) per total volume of liquid or fluid composition (in ml); 'wrt' denotes with respect to; '+ve control' denotes the positive control solution prepared as described above; and '-ve control' denotes the negative control solution prepared as described above.

Table 1

Conditioning Agent	% change in FRA wrt +ve control
Aqueous solution of cetyl trimethyl ammonium chloride (available from Henkel under the trade name 'Dehyquart A')	+380.49 ¹ +389 ²
Dicetyldimonium chloride (available under the trade name 'Adogen 432ET')	1,291
Hydroxypropyl polypeptide C ₁₂ dimethylammonium chloride (available from Croda Chemicals under the trade name 'Croquat L')	2,221
Polyquaternium-39 (available from Chemvicon Speciality Chemicals under the trade name 'Merquat Plus 3330')	+22 ¹ -30 ²
Polyquaternium-6 (available from Chemvicon Speciality Chemicals under the trade name 'Merquat 100')	162
Polyquaternium-7 (available from Calgon Chemicals under the trade name 'Merquat S')	5.52

Footnotes

1 conditioning agent tested as 2% aqueous solution by weight of test substance

2 conditioning agent tested as 2% of test substance in Tris/HCl buffer

It will be noted that solutions comprising the conditioning agents 'Merquat 100', 'Merquat S' or 'Merquat Plus 3330' showed significantly lower free radical activity compared to the other conditioning agents tested.

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The effect of the concentration of various anti-free-radical agents (birch extracts) on the rate of free-radical production (hereinafter referred to as FRP rate) is shown below in Table 2.

Table 2

Anti-free radical agent (Birch Extract)	Concentration (% w/v)	% change in FRP rate wrt +ve control
HP Herbasol Betula in Tris/HCl buffer	1%	-58%
	3%	-69%
	5%	-81%
HP Herbasol Betula in aqueous solution	1%	-43%
	3%	-70%
	5%	-76%
Phytelene of Birch in Tris/HCl buffer	0.1%	-42%
	0.2%	-76%
	0.3%	-84%
	0.4%	-98.5%
	0.5%	-99%
Phytelene of Birch in Tris/HCl buffer (repeated)	0.1%	-57%
	0.2%	-80%
	0.3%	-87%
Aqueous Spray Dried Birch in Tris/HCl buffer	0.1%	-86%
	0.2%	-89%
	0.3%	-90%
Aqueous Spray Dried Birch in Tris/HCl buffer (repeated)	0.025%	-47%
	0.050%	-71%
	0.100%	-85%
	0.150%	-91%
	0.200%	-92%
	0.300%	-92%

- 5 The hair care compositions of the present invention are illustrated by the following non-limiting formulation examples. The components of each formulation example are identified by their chemical name or their CTFA designation. Some of these components are available commercially under the tradenames or designations set out in Table 3.

Table 3

Tradename or designation	Chemical name, description or CTFA designation
Adhesive 28-2930 NAL	Vinyl acetate (VA) / crotonates / vinyl neodeconates copolymer
Aqueous Spray Dried Birch (available commercially from Blagen Chemicals)	Dried maroon powder, flavonoid extract from birch leaves (Folia betulae)
Myacide	Bronopol (2-bromo-2-nitropropane-1,3-diol)
Carbopol 940	carbomer 940
Cromophor RH40	PEG 40 hydrogenated castor oil
Cromophor RH60	PEG 60 hydrogenated castor oil
Euperlan PK 771 (available commercially from Henkel)	Mixture of sodium lauryl ether sulphate (20%), ethylene glycol distearate (20%), cocoyl monoethanolamide (3.5%), laureth-10 (3.5%) and water
Gantrez ES 225	Acrylate / acylamide copolymer
HP Herbasol Betula (available commercially from Cosmetochem UK Ltd)	Hydroglycolic extract (in resp. ratio of 1:2 drug to extract) of birch leaves (Folia betulae)
Hydrosoy 2000	Hydrolysed soya protein (20%) in water
Jaguar	Guar hydroxypropyl trimonium chloride
Merquat 100 (available commercially from Chemiviron Speciality Chemicals)	Polyquaternium-6 (40%) in water

Table 3 (continued)

Tradename or designation	Chemical name, description or CTFA designation
Merquat Plus 3330 (available commercially from Chemiviron Speciality Chemicals)	Polyquaternium-39 (10%) in water
Merquat S (available commercially from Chemiviron Speciality Chemicals)	Polyquaternium-6 (8%) in water
Natrosol 250 NR	Hydroxyethylcellulose
Phytelene of Birch (available commercially from Blagden Chemicals)	Dried bierge powder, flavonoid extract from birch leaves (Folia betulae)
Polawax GP 200 VEG grade (available commercially from Croda Chemicals)	Mixture of cetyl alcohol (80%) and polyethylene glycol (20) stearate
Quaternium 31 (available commercially from Sherex)	Dicetyldimonium chloride
Rowoteric AMB-13 (available commercially from ReWo Chemical Corp)	Mixture of cocoamide propyl betaine (30%), salt (5%) and water
Sequestrene NA2	Disodium ethylene diamine tetracetate dihydrate (disodium EDTA)
Sequestrene NA4	Tetrasodium ethylene diamine tetraacetate (tetrasodium EDTA)
Super Herbasol Extract Birch (available commercially from Cosmetochem (UK) Ltd)	Water and polyene glycol (in respective ratio 60:40) extract from birch leaves (Folia Betulae)
Uvinul MS40	Benzophenone-4

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Formulation Example 1

A pearlised shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (70%) in water	6.38
Mixture of cocoamidopropyl betaine (30%), NaCl (5%) and water	2.85
Polyquaternium-39 (10%) in water	0.5
Mixture of sodium lauryl ether sulphate (20%), ethylene glycol distearate (20%), cocoyl monoethanolamide (3.5%), laureth-10 (3.5%) and water	0.85
2-Bromo-2-nitropropane-1,3-diol	0.02
Pure, vacuum-dried NaCl	2
Citric acid monohydrate	qs
Birch extract (Phytelene of Birch)	0.3
Purified water	to 100

The pearlised shampoo was prepared as follows. The water was slowly added to the sodium laureth-2 sulphate which was stirred. Once the water was mixed in, the remaining ingredients (except the birch extract), were added to the water phase whilst the water phase was stirred. The pH of the resulting mixture was adjusted to between 5 to 6 and then the birch extract was added whilst the mixture was stirred.

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Formulation Example 2

Conditioners were prepared to the following composition:

Component	Concentration(%w/v)
Cetyl alcohol	4
Mixture of cetyl alcohol (80%) and polyethylene glycol (20) stearate (20%)	2
Hydroxyethyl cellulose	1
Polyquaternium-39 (10%) in water	1
Hydrolysed soya protein (20%) in water	0.1
Panthenol (75%) in water	0.05
2-Bromo-2-nitropropane-1,3-diol	0.02
Citric acid	0.02
Birch extract (see below)	as below
Purified water	to 100

The conditioners were prepared as follows. The hydroxyethyl cellulose was dispersed in water at 50C to 60C using a high shear mixer. The pH of the resulting mixture was adjusted as required. The cetyl alcohol and the cetyl alcohol and polyethylene glycol (20) stearate mixture were heated to 60C and then added to the water phase whilst the water phase was mixed with a high shear mixer. The resulting mixture was cooled and the remaining ingredients were added.

10 Free Radical Test Results Examples 2(i) to 2(iv)

Birch extract (% w/v) used in each example	% change FRA wrt +ve control	% change FRA wrt Example 2 without birch extract
2(i) 0.15% Aqueous Spray Dried Birch	- 92	- 94
2(ii) 0.3% Phytelene of Birch	- 91	- 94
2(iii) 3.0% HP Herbasol Betula	- 92	- 94
2(iv) 3.0% SuperHerbasol Extract Birch	- 87	- 91
Example 2 without birch extract	+ 36	-

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Formulation Example 3

A basic shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (70%) in water	6.38
Mixture of cocoamidopropylbetaine (30%), salt (5%) and water	3.84
Polyquaternium-39 (10%) in water	0.5
2-Bromo-2-nitropropane-1,3-diol	0.02
Pure, vacuum-dried NaCl	1.5
Citric acid monohydrate	0.2
Birch extract (Phytelene of Birch)	0.3
Purified water	to 100

The shampoo was prepared by the method used to prepare formulation example 1.

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Formulation Example 4

A leave-in conditioner was prepared to the following composition:

Component	Concentration(%w/v)
D-panthenol (75%) in water	0.75
Polyquaternium-39 (10%) in water	10
Dimethicone propyl PG betaine	0.5
Perfume	0.1
PEG-40 hydrogenated castor oil	0.5
Methylchloroisothiazoline, methylisothiazolinone, magnesium nitrate and water	0.08
Birch extract (Phytelene of Birch)	0.3
Purified water	to 100

The conditioner was prepared as follows. The perfume and the PEG-40 hydrogenated castor oil were stirred together and then the rest of the
5 ingredients were added to this mixture which was stirred.

Free Radical Test Results for Example 4

	% change FRA wrt +ve control	% change FRA wrt standard
Example 4	+120%	-68%
Standard commercially available leave-in conditioner	+588%	-

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Formulation Example 5

A non-aerosol volumising gel spray, designated 'ultra hold', was prepared to the following composition:

Component	Concentration(%w/v)
Denatured alcohol	84.08
Purified water	8.34
PVP / VA copolymer	1.74
PVP	1.2
Isopropyl alcohol	0.6
Rosin acrylate	0.4
Phenyl dimethicone	0.3
Panthenol	0.3
Benzophenone-4	0.02
Polyquaternium-6 (40%) in water	0.05
Birch extract (HP Herbasol Betula)	3

The volumising gel spray was prepared as follows. The PVP/VA copolymer
 5 was dissolved in a mixture of the denatured alcohol and the isopropyl alcohol
 whilst the alcohols were being stirred for 30 minutes. The phenyl dimethicone
 and birch extract were added to this mixture. The benzophenone-4,
 polyquaternium-6, panthenol and PVP were added to the water which was stirred
 to dissolve these ingredients. The resultant aqueous solution was added to the
 10 alcohol mixture whilst the alcohol mixture was stirred.

Free Radical Test Results for Example 5

	% change FRA wrt +ve control	% change FRA wrt Example 5 without polyquaternium 6 or Birch Extract
Example 5	+54%	-53%
Example 5 without polyquaternium-6 or birch extract	+228%	-

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Formulation Example 6

A non-aerosol hairspray, designated 'firm hold', was prepared to the following composition:

Component	Concentration(%w/v)
Denaturated alcohol	86.81
Vinyl acetate / crotonates / vinyl neodecanoate copolymer	4.5
Purified water BP	3.62
PVP / VA copolymer	0.95
Aminomethyl propanol	0.4
Panthenol	0.3
Rosin acrylate	0.2
Benzophenone-4	0.02
Polyquaternium-6 (40%) in water	0.05
Phenyl dimethicone	0.2
Birch extract (HP Herbasol Betula)	3

The hairspray was prepared as follows. The denaturated alcohol and
 5 aminomethyl propanol were stirred together. The remaining ingredients (except the vinyl acetate / crotonates / vinyl neodecanoate copolymer) were added to the ethanol mix and the resultant mixture was stirred. Finally the copolymer was added whilst the mixture was agitated.

Free Radical Test Results for Example 6

	% change FRA wrt +ve control	% change FRA Example 6 without polyquaternium-6 or birch extract
Example 6	-23%	-75%
Example 6 without polyquaternium-6 or Birch extract	-16%	-

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Formulation Example 7

A spray for reviving hair curls was prepared to the following composition:

Component	Concentration(%w/v)
Denatured alcohol	68.23
Purified water (BP)	27.43
PEG-7 glyceryl cocoate	0.4
PVP	0.4
Polyquaternium-4	0.37
Panthenol	0.3
Perfume	0.1
Tocopheryl acetate	0.05
Polyquaternium-39 (10%) in water	0.5
Benzophenone-4	0.02
Methylparaben	0.01
Propylparaben	0
Birch extract (HP Herbasol Betula)	3

The spray was prepared as follows. The polyquaternium-4 was stirred into the water for 30 minutes. The remaining ingredients (except the birch extract
5 and denatured alcohol) were added. Then the denatured alcohol and birch extract were added to the bulk mixture.

Free radical test results for Example 7

	% change FRA wrt +ve control
Example 7	-79%

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Formulation Example 8

A mousse designated 'firm hold' was prepared to the following composition:

Component	Concentration(%w/v)
Propane	3.85
Butane	2.1
PVP / VA Copolymer	1.59
Isobutane	1.05
PVP	0.93
Polyquaternium-4	0.86
Ceteth-20	0.47
C12-13 Pareth-3	0.28
Birch extract (Phytelene of Birch)	0.28
Polyquaternium-11	0.19
Perfume	qs
Panthenol	0.14
Polyquaternium-39 (10%) in water	0.47
Benzophenone-4	0.01
Preservative	qs
Purified water BP	to 100

- The mousse was prepared as follows. The polyquaternium-4 was added to water and the mixture stirred rapidly for 30 minutes. The birch extract was
- 5 pre-dissolved in water and then added to the water mixture. The remaining ingredients were added to this mixture with stirring.

Free radical test results for Example 8

	% change FRA wrt +ve control
Example 8	-5%

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Formulation Example 9

A hair gel was prepared to the following composition:

Component	Concentration(%w/v)
Purified water BP	to 100
PVP	3.5
Carbomer	1
Aminomethyl propanol	0.4
Dimethicone copolyol	0.2
PPG-2 cetareth-9	0.2
Panthenol	0.08
Disodium EDTA	0.05
Quaternium-15	0.05
Polyquaternium-39 (10%) in water	0.5
Benzophenone-4	0.02
Methylparaben	0
Propylparaben	0
Birch extract (Phytelene of Birch)	0.3

The hair gel was prepared as follows. The carbomer was dispersed in two thirds of the water with the disodium EDTA using a high shear mixer. The
 5 aminomethyl propanol was then added to this carbomer dispersion. The rest of the ingredients were added to the remaining water. This water phase mixture was then added to the carbomer dispersion.

Free Radical Test Results for Example 9

	% change FRA wrt +ve control
Example 9	+126%
Standard commercially available hair gel	+473%

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Formulation Example 10

A dry mousse for perming and/or colour-treating hair was prepared to the following composition:

Component	Concentration(%w/v)
Propane	3.85
Butane	2.1
PVP / VA Copolymer	1.3
Isobutane	1
PVP	1
Polyquaternium-4	1
Ceteth-20	0.5
C12-13 Pareth-3	0.3
Birch extract (Phytelene of Birch)	0.3
Polyquaternium-11	0.2
Perfume	0.2
Propylene glycol	0.05
Polyquaternium-39 (10%) in water	0.5
Preservatives	qs
Purified water BP	to 100

The mousse was prepared by the method used to prepare formulation
5 example 8.

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Formulation Example 11

A hair texturising wax was prepared to the following composition:

Component	Concentration(%w/v)
Petrolatum	to 100
Hydrogenated coco-glycerides	5
Paraffin	5
Propylene glycol	1.05
PEG-60 hydrogenated castor oil	1
Purified water BP	0.05
Polyquaternium-39 (10%) in water	0.05
Birch extract (Phytelene of Birch)	0.3
Preservatives	qs

The hair texturising wax was prepared as follows. The propylene glycol and birch extract were stirred together. The PEG-60 hydrogenated castor oil and polyquaternium-39 were stirred into the birch mixture. The remaining ingredients were melted together at 80C, then cooled to 60C and added to the birch mixture.

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Formulation Example 12

A hair-finishing spray was prepared to the following composition:

Component	Concentration(%w/v)
Denatured alcohol	to 100
Butane	27
Isobutane	12
Propane	11
Acrylates / acrylamide copolymer	3
Purified water BP	2
Aminomethyl propanol	0.24
Phenyl dimethicone	0.2
Polyquaternium-6 (40%) in water	0.05
Propylene glycol	1.5
Birch extract (HP Herbasol Betula)	3

The finishing spray was prepared as follows. The alcohol and aminomethyl propanol were stirred together. The acrylates / acrylamide copolymer was
5 slowly added to the alcohol mixture whilst the alcohol mixture was stirred. The phenyl dimethicone, propylene glycol and birch extract were stirred into the mixture and dissolved. The polyquaternium-6 and water were stirred together and added to the bulk mixture.

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Formulation Example 13

A finishing spray, designated 'firm hold', was prepared to the following composition:

Component	Concentration(%w/v)
Denatured alcohol	to 100
Butane	16.2
Isobutane	7.2
Propane	6.6
VA / crotonates / vinyl propionate copolymer	3
Purified water BP	2
Aminomethyl propanol	0.25
Phenyl dimethicone	0.1
Polyquaternium-6 (40%) in water	0.05
Propylene glycol	1.5
Birch extract (HP Herbasol Betula)	3

5 The finishing spray was prepared by an analogous method to that used to prepare formulation example 12 in which the VA / crotonates / vinyl propionate copolymer replaces the acrylate / acrylamide copolymer.

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Formulation Example 14

A pearled shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	20
Cocoamidopropyl betaine	3
Polyquaternium-39 (10%) in water	0.5
Sodium laureth, glycol distearate and cocoamide DEA	2
Viscosity adjuster	qs
Birch extract (Phytelene of Birch)	0.3
pH adjuster	qs
Perfume	0.4
Preservative	qs
Purified water BP	to 100

The pearled shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 15

A shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	20
Cocoamidopropyl betaine	4
Polyquaternium-39 (10%) in water	0.5
Birch extract (Phytelene of Birch)	0.3
Perfume	0.4
Preservative	qs
pH adjuster	qs
Viscosity adjuster	qs
Purified water BP	to 100

The shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 16

A conditioning shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	30
Ammonium lauryl sulphate	20
Cocoamidopropyl betaine	10
Dimethicone propyl PG betaine	0.5
Guar hydroxypropyltrimonium chloride	0.2
Sodium laureth, glycol distearate and cocoamide DEA	2
Polyquaternium-39 (10%) in water	2.5
Perfume	qs
Birch extract (Phytelene of Birch)	0.3
pH adjuster	qs
Preservatives	qs
Purified water BP	to 100

The conditioning shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 17

A conditioning shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	30
Ammonium lauryl sulphate	20
Cocoamidopropyl betaine	10
Dimethicone propyl PG betaine	0.5
Guar hydroxypropyltrimonium chloride	0.2
Sodium laureth, glycol distearate and cocoamide DEA	2
Polyquaternium-39 (10%) in water	2.5
Perfume	0.4
Birch extract (HP Herbasol Betula)	3
pH adjuster	qs
Preservatives	qs
Purified water BP	to 100

The conditioning shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 18

A conditioning shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	30
Ammonium lauryl sulphate	20
Cocoamidopropyl betaine	10
Dimethicone propyl PG betaine	0.5
Guar hydroxypropyltrimonium chloride	0.2
Sodium laureth, glycol distearate and cocoamide DEA	2
Polyquaternium-39 (10%) in water	2.5
Perfume	0.4
Birch extract (Aqueous Spray Dried Birch)	0.15
pH adjuster	qs
Preservatives	qs
Purified water pH	to 100

The conditioning shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 19

A hair mask with active mud was prepared to the following composition:

Component	Concentration(%w/v)
Molecular sieve	35
Hydrogen vegetable protein	7
Cetearyl alcohol and PEG-23 stearate	5
Cetyl alcohol	3
Cocamide MEA	3
Stearamidopropyl dimethylamine	3
Polyquaternium-39 (10%) in water	1
Fragrance	0.5
Birch extract (Phytelene of Birch)	0.3
Propylene glycol	to 100

The hair mask was prepared as follows. The cetearyl alcohol and PEG-23 stearate mixture, the cetyl alcohol and the propylene glycol were heated
5 together to 70C. This mixture was mixed until homogenous and the rest of the ingredients added and homogenously mixed together. The composition was cooled whilst stirring.

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Formulation Example 20

A hair serum was prepared to the following composition:

Component	Concentration(%w/v)
Alcohol, t-butyl alcohol and denatonium benzoate	25
Polyquaternium-39 (10%) in water	1
Carbomer 940	0.6
Fragrance solubiliser	qs
Fragrance	0.5
Birch extract (Phytelene of Birch)	0.2
Tetrasodium EDTA	0.04
2-Bromo-2-nitropropane-1,3-diol	qs
pH Adjuster	qs
Purified water BP	to 100

The hair serum was prepared as follows: The carbomer 940 was dispersed and hydrated in half of the water. The pH of 30% of the water was adjusted to about pH 5.2 and the birch extract was added. This birch mixture was added to a high shear mixer and mixed until uniform. The birch mixture was then added to the carbomer mixture. The fragrance solubiliser was warmed and mixed with the fragrance and this mixture was added to the bulk birch / carbomer mixture. When the resultant mixture was thoroughly mixed the remaining ingredients were added (the 2-bromo-2-nitropropane-1,3-diol was previously dissolved in purified water before adding to the mixture at this stage). The serum was made up to the required volume with purified water.

Formulation Example 21

A hair rebuilder was prepared to the following composition:

Component	Concentration(%w/v)
Guar hydroxypropyltrimonium chloride	0.3
Alcohol, t-butyl alcohol and denatonium benzoate	25
Fragrance	0.5
Fragrance solubiliser	qs
Birch extract (Phytelene of Birch)	0.2
Polyquaternium-39 (10%) in water	1
Panthenol and water	1
pH Adjuster	qs
Purified water BP	to 100

- The hair rebuilder was prepared as follows. The guar hydroxypropyltrimonium chloride was dispersed in 20% of the purified water.
- 5 The pH of 30% of the water was adjusted to about pH 5.2 and the birch extract was added. This birch mixture was then mixed in a high shear mixer until uniform. The birch mixture was then added to the guar hydroxypropyltrimonium chloride mixture. The fragrance solubiliser was warmed and mixed with the fragrance. This mixture was added to the bulk birch mixture and the resulting
- 10 mixture was stirred together until well mixed. The remaining ingredients were added and the rebuilder was made up to the required volume with purified water.

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Formulation Example 22

A shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth sulphate and Water	14
Cocamide DEA and DEA-cocoate	1.5
Cocamidopropyl betaine and salt	8
Lauramide DEA	1.5
Birch extract (Phytelene of Birch)	0.2
Polyquaternium-39 (10%) in water	1
Panthenol and water	1
Fragrance	0.5
Preservative	qs
Fragrance solubiliser	0.5
Fatty acid partial glyceride polyglycol ether and ester	4
pH adjuster	qs
Viscosity adjuster	qs
Purified water BP	to 100

The shampoo was prepared by the same method used to prepare formulation example 1.

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Formulation Example 23

A conditioner was prepared to the following composition:

Component	Concentration(%w/v)
Birch extract (Phytelene of Birch)	0.2
Polyquaternium-39 (10%) in water	1
Hydrolysed vegetable protein	7
Stearamidopropyl dimethylamine	0.5
Propylene glycol	0.5
Dicetyldimonium chloride, ethanol and water	3
Stearyl alcohol and ceteareth-20	1
Cetyl alcohol	3.5
Potassium chloride	0.3
Cyclomethicone	1.8
Perfume	0.3
2-Bromo-2-nitropropane-1,3-diol	qs
Viscosity adjuster	qs
pH adjuster	qs
Purified water BP	to 100

The conditioner was prepared as follows. The birch extract was pre-dissolved in water and added to 80% of the water. The solution was mixed
5 in a high shear mixer until uniform, and then heated to 80C. The cetyl alcohol and the stearyl alcohol and ceteareth-20 mix were heated to 80C and mixed together with the water phase using the high shear mixer. This mixture was cooled whilst being stirred and when the mixture temperature fell below 30C, the remaining ingredients were added. The product was made up to the
10 required volume with purified water.

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Formulation Example 24

A leave-in conditioner was prepared to the following composition:

Component	Concentration(%w/v)
Guar hydroxypropyltrimonium chloride	0.3
Alcohol, t-butyl alcohol and denatomium benzoate	25
Fragrance solubiliser	qs
Fragrance	0.5
Birch extract (Phytelene of Birch)	0.2
Polyquaternium-39 (10%) in water	1
pH adjuster	qs
Purified water BP	to 100

The conditioner was prepared as follows. The pH of 40% of the water was adjusted to between pH 3.0 and 3.5 and this pH adjusted water was then
5 heated to between 60C and 70C. The birch extract was added to this heated water whilst being stirred rapidly and the stirring continued until the solution was clear. A further 40% of the water was added to this solution, followed by the guar hydroxypropyltrimonium chloride whilst the water was stirred rapidly for 15 minutes or until the solution became clear. The solution was cooled to below
10 30C and the remaining ingredients were added.

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Formulation Example 25

A shampoo was prepared to the following composition:

Component	Concentration(%w/v)
Sodium laureth-2 sulphate (27%)	20
Cocoamidopropyl betaine	4
Polyquaternium-7 (8%) in water	0.2
Birch extract (Phytelene of Birch)	0.3
Perfume	0.4
Viscosity adjuster	qs
pH adjuster	qs
Preservative	qs
Purified Water BP	to 100

The shampoo was prepared by the method used to prepare formulation example 1.

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Formulation Example 26

A hair gel was prepared to the following composition

Component	Concentration(%w/v)
Purified water BP	to 100
Polyvinylpropionate	3.5
Carbomer	1
Aminomethyl propanol	0.4
Dimethicone copolyol	0.2
PPG-2 Ceteareth-9	0.2
Panthenol	0.08
Disodium EDTA	0.05
Quaternium-15	0.05
Polyquaternium-7 (8%) in water	0.5
Benzophenone-4	0.02
Methylparaben	0
Propylparaben	0
Birch extract (Phytelene of Birch)	0.3

The hair gel was prepared by the method used to prepare formulation example 9.

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Formulation Example 27

A conditioner was prepared to the following composition:

Component	Concentration(%w/v)
Birch extract (Phytelene of birch)	0.2
Polyquaternium-7 (8%) in water	0.5
Stearamidopropyl dimethylamine	0.5
Propylene glycol	0.5
Dicetyldimonium chloride, and ethanol and water	3
Stearyl alcohol and cetareth-20	1
Cetyl alcohol	3.25
Potassium chloride	0.3
Cyclomethicone	1.8
Perfume	0.3
Preservative	qs
pH adjuster	qs
Purified water BP	to 100

The conditioner was prepared by the same method used to prepare formulation example 23.

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Formulation Example 28

Leave-in conditioners were prepared to the following composition:

Component	Concentration(%w/v)
Panthenol (75%) in water	0.9
Denatured ethanol	25.33
Polyquaternium 39 (10%) in water	0.75
Octyl dimethyl p-aminobenzoate acetate	0.1
Propylene glycol	0.86
PEG-40 hydrogenated castor oil	0.25
2-Bromo-2-nitropropane-1,3-diol	0.02
Birch extract (see below)	as below
Purified water BP	to 100

The leave-in conditioners were prepared as follows. The pH of 40% of the water was adjusted to between 3.0 and 3.5, and this pH adjusted water was heated to between 60C and 70C. The birch extract was added to the heated stirred water, which was stirred until the mixture became clear. A further 40% of the water was added and the solution was cooled to below 35C when the remaining ingredients were added.

Free Radical Test Results for Examples 28(i) to 28(ii)

Birch extract (% w/v used in the example)	% change FRA wrt +ve control	% change FRA wrt Example 28 without birch extract
28(i) 3% HP Herbasol Betula	- 65.5	- 86
28(ii) 3% Super Herbasol Extract Birch	- 84.5	- 94
Example 28 without birch extract	143	-

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Formulation Example 29

A conditioner was prepared to the following composition:

Component	Concentration(%w/v)
Cetyl alcohol	4
Cetostearyl alcohol and PEG-20 stearate	2
Hydroxyethylcellulose	1
Polyquaternium-39 (10%) in water	1
D-panthenol (75%)	0.05
2-Bromo-2-nitropropane-1,3-diol	0.02
Methylchloroisothiazolinone, Methylisothiazolinone, magnesium nitrate and water	0.05
Citric acid	0.02
Birch extract (Phytelene of Birch)	0.3
Perfume	0.3
Purified water BP	to 100

The conditioner was prepared as follows. The hydroxyethylcellulose was hydrated in 80% of the water. The birch extract was pre-dissolved in water and added to the hydroxyethylcellulose mixture which was then mixed in a high shear mixer until uniform and then heated to 80C. The cetyl alcohol and the cetostearyl alcohol and PEG-20 stearate mix were heated to 80C and mixed together with the cellulose mixture using the high shear mixer. The resulting mixture was cooled whilst being stirred and when the mixture temperature fell below 30C the remaining ingredients were added. The product was made up to the required volume with purified water.

Free Radical Test Results for Example 29

	% change FRA wrt +ve control
Example 29	-81%
Standard commercially available conditioner	-12.5%

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CLAIMS

1. A hair care composition comprising an anti-free-radical agent and a polymeric conditioning agent prepared from monomer entities selected from one or more of: acrylic acid, acrylamide, dimethyldiallylammonium chloride and
5 any mixtures thereof.
2. A composition as claimed in claim 1, in which the anti-free-radical agent is selected from one or more of: ascorbic acid, tocopherol a herbal extract and any mixtures thereof.
3. A composition as claimed in claim 2, in which the herbal extract is a birch
10 extract.
4. A composition as claimed in any preceding claim with reference to the examples.
5. A method of preparing a hair care composition comprising mixing in a suitable manner an anti-free radical agent with a polymeric conditioning agent
15 prepared from monomer entities selected from one or more of acrylic acid, acrylamide, dimethyldiallylammonium chloride and any mixtures thereof.
6. A method of preparing a hair care composition as described herein with reference to the examples.
7. A method of treating hair by application of a composition as claimed in any
20 of claims 1 to 4.
8. A method as claimed in claim 7, in which the hair treatment is selected from one or more of: washing, conditioning and styling.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 96/02135

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A61K7/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 35 34 287 A (WELLA AG) 2 April 1987 see page 5, line 10 see claims 1-12 -----	1

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Ir. Information on patent family members

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